
वस्त्रादि — नाभिकीय जैविक रासायनिक
(एन बी सी) पारगम्य सुरक्षात्मक वस्त्र
भाग 1 रासायनिक अपशिष्ट तत्व के अनावरण पर
भेदन समय ज्ञात करने की गुणात्मक विधि —
सल्फर मस्टर्ड (एचडी)

**Textiles — Nuclear Biological
Chemical (NBC) Permeable
Protective Clothing**

**Part 1 Qualitative Method of Determining
Breakthrough Time on Exposure to Chemical
Warfare Agent — Sulfur Mustard (HD)**

ICS 13.340.10

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Textiles Protective Clothing Sectional Committee had been approved by the Textile Division Council.

Among the Weapons of Mass Destruction (WMD), Chemical Warfare Agents (CWAs) are probably one of the most brutal created by mankind. CWAs are extremely toxic synthetic chemicals that can be dispersed as a liquid, gas, vapour or aerosol. These CWAs have either lethal or incapacitating effects on humans. Thousands of toxic substances are known, but only some of them are considered as CWAs based on their characteristics, namely, high toxicity, imperceptibility to senses, rapidity of action after dissemination, persistency are listed as scheduled chemicals in the Chemical Weapons Convention (CWC). The CWAs possess different characteristics and belong to various classes of compounds with pronounced physicochemical properties. Thus, they are classified in many ways. Based on their volatility, they are classified as persistent, semi-persistent and non-persistent agents. Agents like, phosgene, hydrogen cyanide, sarin, soman, cyclo sarin and tabun are non-persistent agents whereas the less volatile agents like sulfur mustard (HD) and VX are persistent agents. Based on their chemical structure, they can be classified as organophosphorus (OP), organosulfur, organofluorine and arsenicals. In general, classifications in terms of physiological effects produced on humans by the CWAs have been used for many decades. Thus, the CWAs used in warfare are classified as follows:

- a) Nerve agents,
- b) Vesicants (blistering agents),
- c) Blood agents (cyanogenic agents),
- d) Choking agents (pulmonary agents),
- e) Riot-control agents (tear gases),
- f) Psychomimetic agents, and
- g) Toxins.

Defence Research and Development Establishment (DRDE) is the nodal laboratory of DRDO working on the various aspects of detection, protection and decontamination technologies against chemical and biological warfare agents. It has Organization for Prohibition of Chemical Weapon (OPCW) declared Schedule 1 facility to synthesize and use CWAs for R and D activities, therefore DRDE has the mandate to establish test and evaluation facilities for the Chemical-Biological threat mitigation technologies. In this context, a facility to evaluate the materials used in protective gears for the permeation of CWAs has been established at DRDE, Gwalior. The facility and the standard would be beneficial to the R and D organizations and industries for the comparison and suitability of materials to be used in various protection technologies depending upon their specific requirements.

Performance evaluation of protective clothing is carried out by assessment of the performance in the simulated test environment which is created by CWAs in liquid/vapour form. In case of liquid/vapour challenge under laboratory conditions, the test environment is created by release of definite concentration of a particular agent under specified temperature and humidity conditions. The outcome of the testing will ensure the performance evaluation of permeable protective clothing.

This standard covers the qualitative tests of permeable protective clothing against HD, which includes HD-BTT test, mandrel test, expulsion test and inverted expulsion test.

In the formulation of this standard, technical assistance has been derived from the following standards/publications:

UK/SC/3346G — Requirements for a non-woven cloth coated on one side with activated charcoal

MIL-DTL-32102 — Joint service lightweight integrated suit technology (JSLIST) coat and trouser, chemical protective clothing

Test operations procedures (TOP) 08-02-501 — Permeation testing of materials with chemical agents or simulants (Swatch testing)

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

TEXTILES — NUCLEAR BIOLOGICAL CHEMICAL (NBC) PERMEABLE PROTECTIVE CLOTHING

PART 1 QUALITATIVE METHOD OF DETERMINING BREAKTHROUGH TIME ON EXPOSURE TO CHEMICAL WARFARE AGENT — SULFUR MUSTARD (HD)

1 SCOPE

1.1 This standard (Part 1) covers the detailed procedures for test and evaluation of permeable protective clothing against sulfur mustard (HD).

1.2 This standard specifies qualitative test methods to determine break through time of chemical warfare agent (CWA) challenge through swatches.

1.3 It specifies the methods for HD breakthrough time (HD-BTT), mandrel, expulsion and inverted expulsion testing of swatches which gives indication of chemical resistance against HD.

1.4 This standard provides relative ranking or material screening information about the ability of test materials to resist against HD breakthrough.

1.5 This standard shall be employed as a quality control (QC) measure for developmental, pilot, bulk production and stored samples/lots.

1.6 This standard does not cover any information regarding toxic industrial chemicals (TICs) and toxic industrial materials (TIMs).

1.7 The definitions in this standard apply only to this specification and shall be the determining factor(s) when interpreting any word or combination(s) of words.

1.8 This standard does not cover all the associated safety concerns for handling and testing of protective clothing against HD. It is the responsibility of the user to establish appropriate safety and health practices to determine the applicability of regulatory limitations prior to use.

1.9 Nothing in this standard prevents to have higher level of protection for handling of CWAs.

1.10 These tests shall not only be taken as measure of the protection time given by the tested material under actual used conditions.

1.11 This standard alone is not sufficient to assess the ability of whole ensembles made from tested materials

to protect the wearer. These procedures are designed to be used as part of an overall assessment program, evaluating the material performance, manufacturing and integration with other pieces of the protective ensemble.

2 REFERENCES

The standard listed below contains provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

<i>IS No.</i>	<i>Title</i>
6359 : 1971	Method for conditioning of textiles

3 TERMS AND DEFINITIONS

For the purpose of this standard the following definitions shall apply.

3.1 Adsorption — A process in which molecules are concentrated on a surface by chemical or physical forces, or both.

3.2 Breakthrough — A mass or concentration of a challenge contaminant found on the initially clean side of a contaminated test swatch, indicating that the contaminant has permeated or penetrated the material being tested.

NOTE — Breakthrough threshold levels are defined for each test program and shall be specified as needed in the test plan or other test planning documents.

3.3 Breakthrough Time — It is the time interval in minutes between placement of HD on the test swatch sample and the first indication of appearance of blue dot on congo red paper (detector paper).

3.4 Challenge — The chemical agent or agent simulant (or other chemical compound) that is placed on one

side of a test swatch. The challenge can be in liquid or vapour form. The challenge concentration is expressed in units of g/m² for liquid or mg/m³ for vapour.

3.5 Chemical Warfare Agents (CWAs) — CWA is a chemical substance whose toxic properties are used to kill, injure or incapacitate human beings.

3.6 Decontamination — The process of making any person, object, or area safe by adsorbing, destroying, neutralizing, making harmless, or removing toxic chemicals to or around it.

3.7 Penetration — The flow of a chemical through closures, pores, seams, pinholes, or imperfections in a protective clothing material on a non-molecular level.

3.8 Permeation — The process by which a chemical moves through a protective clothing material on a molecular level. Permeation involves the following:

- sorption of molecules of the chemical into the contacted surface (usually the outside) of a material,
- diffusion of the sorbed molecules among the material molecules, and
- desorption of the molecules from the non-contacted surface (usually the inside) of the material into the collecting medium.

3.9 Sulfur Mustard (HD) — A chemical warfare agent belongs to the class of blister agent (vesicant), that causes severe burns on exposure to the skin, eyes and respiratory tract.

3.10 Swatch — A small sample, usually taken from test specimen.

3.11 Test Control Swatch — Both positive and negative test control swatches shall be used in each trial. The negative control swatch shall be a swatch taken from butyl rubber that is conditioned the same way as the test swatches. The positive control swatch shall have well defined permeation profile which is not resistant to CWAs.

3.12 Test Specimen — All the layers of fabric or other materials arranged in the order and orientation as used in practice while wearing the chemical protective clothing.

4 PRINCIPLE

4.1 Sulfur Mustard – Break through Time (HD-BTT) Test

It is qualitative way to determine the HD penetration through fabric at 20 ± 1°C temperature at ambient pressure. The test is based on the generation of HCl gas by the reaction of HD with chlorinating reagent that is spot disc (SD) reagent which changes the

colour of congo red paper from pink to blue because of acidic nature. Break through time against HD shall be measured as the first appearance of blue spot on the congo red paper.

4.2 Mandrel Test

It is qualitative way to determine the HD penetrated through carbon coated fabric / protective clothing under stress for one hour duration under ambient temperature and pressure. The HD penetration is ascertained by using three colour detector paper (TCD). TCD paper works on dissolution principle, as different CWAs dissolve different dyes and gives its respective colour. Break through time against HD shall be measured as the first appearance of orange-red spot on the TCD paper.

4.3 Expulsion and Inverted Expulsion Test

It is qualitative way to determine the HD penetrated through carbon coated fabric/ protective clothing under pressure for one hour duration under ambient temperature and pressure. The HD penetration is ascertained by using TCD. Break through time against HD shall be measured as the first appearance of orange-red spot on the TCD paper.

5 CHEMICALS AND TEST SET UP

5.1 Sulfur Mustard Break through Time (HD-BTT)

5.1.1 Chemicals

- HD shall be >99 percent purity (purity shall be determined with ¹H-NMR).
- Chlorinating Reagent — SD Reagent (2, 4 dichlorophenyl benzoyl chloroimide) active chlorine (8-11 percent).

5.1.2 Test Set Up

- Calibrated micropipette — (0.2-20 µl).
- HD BTT chamber (*see* Fig. 1).
- Brass/stainless steel disc (50 mm diameter).
- Spacer ring/small flange, brass/stainless steel — 53 mm diameter with an inner hole of 19 mm diameter.
- Large flange, brass/stainless steel — 1 mm thick, 57 mm diameter with an inner hole of 32 mm in the centre.
- Glass plate — 64 mm × 64 mm and 9.5 mm thick.
- Congo red paper (detector paper) — 38 mm diameter.

5.2 Mandrel Test

5.2.1 Chemicals

- HD shall be >99 percent purity (purity shall be determined with ¹H-NMR).

5.2.2 Test Set Up

- a) Calibrated micropipette — (01-10 µl)
- b) Mandrel test apparatus consisting of components as shown in Fig. 2
- c) Mirror
- d) Glass tube (41 mm internal diameter, 45 cm length)
- e) Three color detector paper (TCD)
- f) Weight — 234 g made up of stainless steel 316 grade.

5.3 Expulsion and Inverted Expulsion**5.3.1 Chemicals**

- a) HD shall be >99 percent purity (purity shall be determined with ¹H-NMR).

5.3.2 Test Set Up

- a) Calibrated Micropipette — (01-10 µl).
- b) Expulsion and inverted expulsion test set up consisting of the components as shown in Fig. 3 and Fig. 4 respectively.
- c) Glass plate.
- d) Three colour detector paper (TCD).
- e) Weight(s) — 70.2 g made up of stainless steel 316 grade.

6 SAFETY

HD is a chemical warfare agent. Protective gears must be used by the individual while handling HD. The test shall be performed in a fume hood.

NOTE — These safety precautions are for information purpose only. The precautions are ancillary to the testing procedures and are not intended to be all inclusive. Good laboratory practices shall be followed.

7 SAMPLING**7.1 HD-BTT**

7.1.1 The swatch samples shall have the dimensions 50 mm diameter and shall be taken at random places of the test specimen.

7.1.2 Ensure test specimens are free of folds, crease or wrinkles. Avoid the contact of oil, water and grease with the specimens during handling.

7.1.3 The swatch samples shall be marked out.

7.2 Mandrel Test

7.2.1 Test swatch samples of $5 \times 15 \text{ cm}^2$ shall be drawn randomly from each individual piece of the test specimen.

7.2.2 Ensure test specimens are free of folds, crease, or wrinkles. Avoid the contact of oil, water and grease with the specimens during handling.

7.2.3 The swatch samples shall be marked out.

7.3 Expulsion and Inverted Expulsion

7.3.1 Swatch samples of 5.1 cm diameter (or $5.1 \times 5.1 \text{ cm}^2$) shall be drawn at random from each individual piece of the test specimen.

7.3.2 Ensure test specimens are free of folds, crease, or wrinkles. Avoid the contact of oil, water and grease with the specimens during handling.

7.3.3 The swatch samples shall be marked out.

7.4 Number of Swatch Samples

A minimum of six swatch samples shall be tested for each test material other than one positive control sample and negative control sample. Each test shall have two sets of control swatches, positive and negative. Positive control swatch sample shall have well defined permeation profile which is non resistant to CWAs. Negative control swatch shall be taken from butyl rubber.

8 CONDITIONING AND TESTING ATMOSPHERE**8.1 Conditioning Atmosphere**

Prior to testing, the specimens shall be conditioned for at least 24 h at a temperature of $(27 \pm 2)^\circ\text{C}$ and a relative humidity of 65 ± 5 percent (*see* IS 6359). If testing is not carried out immediately after conditioning, place the conditioned test specimens in a sealed container.

8.2 Testing Atmosphere

Perform the tests in ambient atmosphere except HD-BTT test. HD-BTT test shall be performed at $20 \pm 1^\circ\text{C}$ temperature.

9 TEST PROCEDURE**9.1 HD-BTT Test**

9.1.1 Switch on the HD-BTT test chamber.

9.1.2 Take a brass/stainless steel disc having dimension of 50 mm diameter.

9.1.3 Place a white cotton drill fabric of 1 cm^2 on the brass/stainless steel disc. Put spacer ring/small flange of 53 mm diameter with an inner hole of 19 mm diameter on the cotton drill fabric. The purpose of the flange is to retain the white cotton cloth on the brass/stainless steel disc.

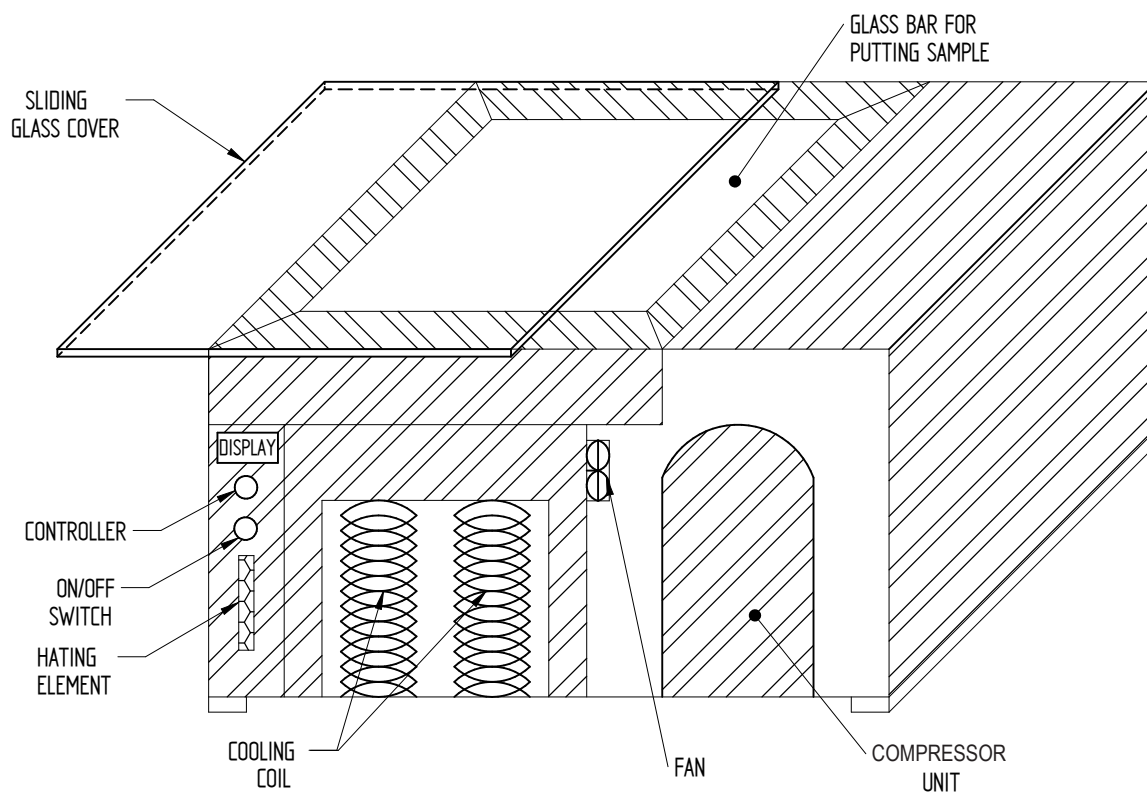


FIG. 1 HD-BTT TEST CHAMBER

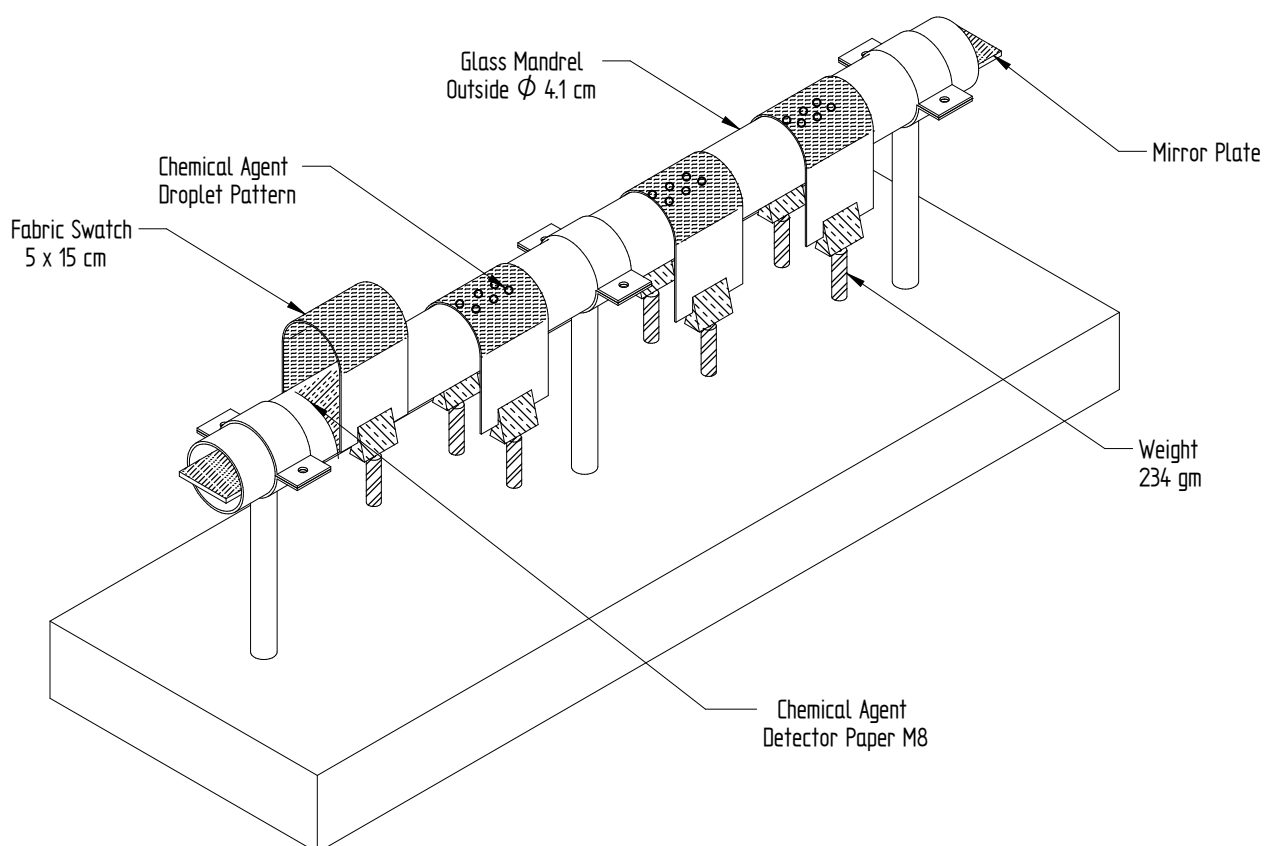


FIG. 2 MANDREL TEST APPARATUS

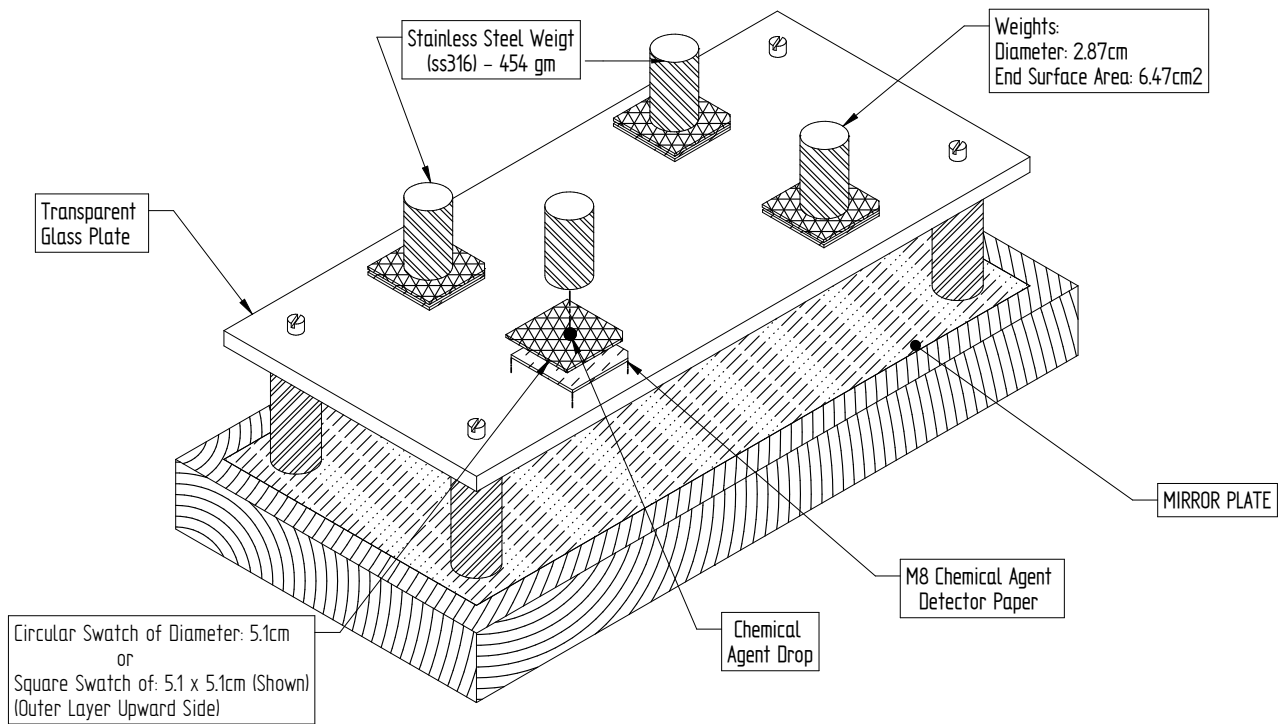


FIG. 3 EXPULSION TEST SET UP

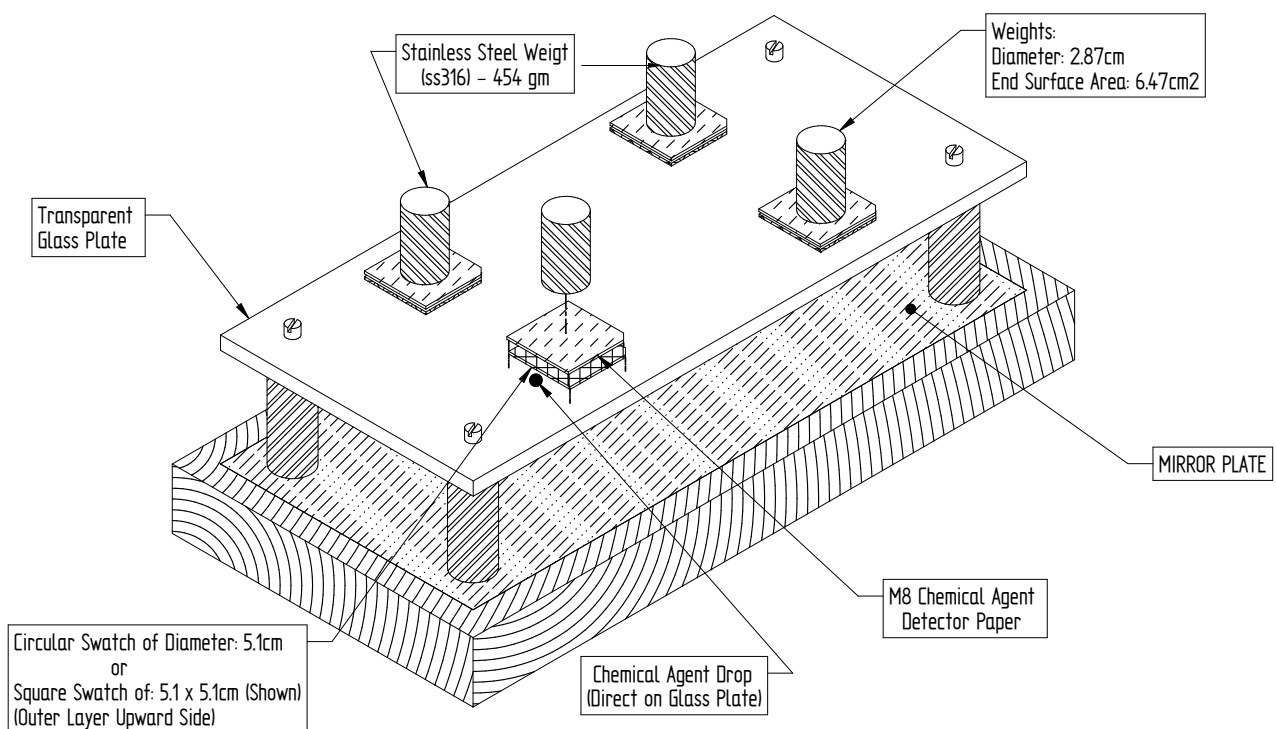


FIG. 4 INVERTED EXPULSION TEST SET UP

9.1.4 On the exposed cloth visible from 19 mm hole of flange, drop 12 μL (15 mg) of HD agent using calibrated micropipette.

9.1.5 The spacer ring shall be temporarily covered with glass plate for 30 min in the fume hood and the whole assembly shall be kept in a test chamber maintained at $20 \pm 1^\circ\text{C}$.

9.1.6 To prepare the swatch sample, 2 pieces of 50 mm diameter shall be taken and these are placed in such a way that adsorbent sides (carbon/charcoal/activated carbon sphere) shall be facing each other. On the top of the swatch samples, place the congo red paper of 38 mm diameter spotted with SD reagent with the help of micropipette tips.

9.1.7 The swatch sample and congo red paper are to be laid down on the large flanged brass in the following sequence: Congo red paper upper most and retained in the place by means of a large flanged brass 1 mm thick, 57 mm internal diameter at the flange with a hole of 32 mm in the centre. Finally a piece of clean glass plate (64 mm $\times$ 64 mm) is placed on top of the large flanged brass, the whole assembly shall be placed in HD-BTT chamber without delay. The whole assembly shall be left undisturbed throughout the period of test.

9.1.8 The congo red paper is examined through the glass plate at regular interval of time to identify the break through time by visual check of appearance of blue spot. The HD penetration time is taken as the interval in minutes between laying the swatch sample discs on the spacer ring and the first indication of blue spots on the congo red paper.

9.2 Mandrel Test

9.2.1 Swatch sample of $5 \times 15 \text{ cm}^2$ shall be placed evenly over TCD paper on round glass mandrel so that the outer surface of the swatch faces upwards.

9.2.2 The swatch sample shall be stressed by hanging 234 g weight each at two ends (total weight 468 g) of swatch sample.

9.2.3 Six drops, each of 4 μL of HD shall be applied to the surface of the swatch sample.

9.2.4 The TCD paper shall be examined periodically till an hour after HD application for evidence of HD penetration.

9.2.5 If visible evidence of HD agent penetration is seen before 1 h, the time at which the HD penetration became visible shall be recorded and the test shall be taken as complete. The test shall end after 1 h if no penetration is observed before that time.

9.3 Expulsion Test

9.3.1 Swatch sample of 5.1 cm diameter (or $5.1 \times 5.1 \text{ cm}^2$) shall be placed evenly over TCD paper.

9.3.2 TCD paper shall be kept on the glass plate facing sensing side of the paper in upward direction followed by placement of swatch sample outer surface facing upward.

9.3.3 A single HD drop of 4 μL shall be applied to centre of the swatch sample.

9.3.4 After a period of 15 s, the TCD paper shall be examined for a sign of penetration of HD.

9.3.5 If no penetration is visible, a pressure of 70.2 g/cm^2 shall be applied to the swatch for 1 h.

9.3.6 The TCD paper shall be examined periodically till an hour after HD application for evidence of HD penetration.

9.3.7 If visible evidence of HD penetration is seen before 1 h, the time at which the HD penetration became visible shall be recorded and the test shall be taken as complete. The test shall end after 1 h if no penetration is observed before that time.

9.4 Inverted Expulsion Test

9.4.1 Swatch sample 5.1 cm diameter (or $5.1 \times 5.1 \text{ cm}^2$) shall be placed evenly over TCD paper.

9.4.2 Then, a single HD drop of 4 μL shall be placed on a flat glass plate to spread for 1 min.

9.4.3 Swatch sample shall be placed on the drop of the HD in such a way that the swatch outer surface faces downwards on the top of drop and TCD paper faces upwards.

9.4.4 A pressure of 70.2 g/cm^2 shall be applied to the swatch for 1 h.

9.4.5 The TCD paper shall be examined periodically till an hour for evidence of HD penetration.

9.4.6 If visible evidence of HD agent penetration is seen before 1 h, the time at which the HD penetration became visible shall be recorded, and the test shall be taken as complete. The test shall end after 1 h if no penetration is observed before that time.

10 DECONTAMINATION

After execution of the test using HD all the material shall be decontaminated as per the following procedure:

10.1 One container/bucket (20 litre capacity) with lid, filled with freshly prepared 10-12 percent sodium

hypochlorite solution or any suitable decontaminant shall be kept in the fuming hood.

10.2 After completion of the experiment, all used glasswares, tubes, joints, tubing, parafilms, sample swatches and SS chambers shall be put in the bucket of decontamination solution and left for 24 h in the fuming hood.

10.3 After 24 h, 50 ml solution from the bucket shall be taken and extracted 3 times with 20 ml chloroform and combined extracts (60 ml) shall be concentrated up to 0.5 ml.

10.4 This concentrated extract shall be analyzed by gas chromatography (GC) to ensure the complete degradation of HD.

10.5 If the results of GC showed the incomplete decontamination, then again fresh solution of sodium hypochlorite shall be added in the bucket.

10.6 It shall be continued till the GC showed complete decontamination of the HD.

10.7 Finally the decontaminated swatch samples of chemical protecting clothing shall be removed

from the decontamination solution and further incinerated.

11 TEST REPORT

The test report shall contain the particulars as mentioned below:

- a) Name of the test laboratory;
- b) Test report number;
- c) Date;
- d) Test standard;
- e) Reference letter;
- f) Environmental conditions;
- g) Equipment under evaluation with serial number/identification;
- h) Report prepared by, reviewed by and approved by;
- j) Test witnessed by ;
- k) Period of testing ;
- m) Number of pages ; and
- n) Test result format, as given in Table 1.

Table 1 Test Result Format

(Clause 11)

Sl No.	Sample No.	Test	Specified Requirement	Test Standard	Observed Value Hours : Minutes	Remarks

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Textiles Protective Clothing Sectional Committee, TXD 32

<i>Organization</i>	<i>Representative(s)</i>
Northern India Textile Research Association, Ghaziabad	DR ARINDAM BASU (Chairman) DR M. S. PARMAR (<i>Alternate</i>)
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Indian Institute of Technology, New Delhi	REPRESENTATIVE

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Teijin India Private Limited, Gurugram	SHRI PRIYARPAN SRIVASTAVA SHRI RAVI KUMAR (<i>Alternate</i>)

IS 17377 (Part 1) : 2020

<i>Organization</i>	<i>Representative(s)</i>
Tex Corporation Limited, Gurugram	SHRI VIJAY TOLEY SHRI SANJAY AGGARWAL (<i>Alternate</i>)
Textiles Committee, New Delhi	SHRI PARVEJ AHMED SHRI V. K. R. MACHIRAJU (<i>Alternate</i>)
TUV Rheinland India Pvt Ltd, Gurugram	SHRI CHARAN SINGH
In Personal Capacity	DR V. K. KOTHARI
BIS Directorate General	SHRI A. K. BERA, SCIENTIST 'F' AND HEAD (TXD) [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

Member Secretary

SHRI J. K. GUPTA
SCIENTIST 'D' (TXD), BIS

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402

Website: www.bis.gov.in

Regional Offices:

	Telephones
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	{ 2323 7617 2323 3841
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